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SPACE PROCESSING APPLICATIONS OF ION BEAM TECHNOLOGY

FINAL REPORT

15 August 1977

Contract NAS3-20095

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FOREWORD

This document is the final report for Contract NAS3-20095 and was prepared for the National Aeronautics and Space Administration, Lewis Research Center, by personnel of Lockheed Missiles & Space Company, Huntsville Research & Engineering Center, Huntsville, Alabama. Benard Sater of NASA-Lewis was the contract monitor for the last part of the study and Robert Vetrone, also of NASA-Lewis, for the first part of the study.

ACKNOWLEDGMENTS

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INTRODUCTION AND SUMMARY

Recent developments in two different technological areas raise the possibility that the two seemingly unrelated areas could have a mutually beneficial interaction. The two technologies under discussion are: (1) the development of ion thruster engines for spacecraft propulsion and maneuvering, and (2) the development of material processing operations for space environments. The possible association of these two technologies becomes evident when it is appreciated that ion thruster engines can readily serve as ion beam sources. Ion beam technology is growing at a rapid rate on earth and is finding a number of commercial applications. The present study, therefore, addressed itself to the identification of potential space processing applications of thruster ion beam (TIB) technology. The criterion for a potential space processing application was necessarily that the space environment offered some advantage — either economic or unique capability — over that afforded by the earth environment. Two aspects of the effects of space environments on TIB in general were considered: possible advantages of space vacuum environments and possible gravity effects on TIB materials operations such as thin film growth, ion milling, and surface texturing.

A consideration of the various factors involved in ion beam generation and processing leads to the conclusion that the almost limitless pumping capacity of space will allow easy maintenance of large pressure differentials and hence optimal conditions for ion beam generation and UHV conditions for thin film deposition. Limitless pumping capacity will further allow large-scale utilization of ion beams for operations such as microelectronic component fabrication. Such possibilities will result in product quality improvement at lower cost.

In another part of the study, experiments on both TIB sputter deposition and vapor deposition at one atmosphere and at 1 torr (133 Pa) were conducted. The results indicate that a direct gravity effect may be involved in thin film

deposition processes even at high vacuums. A direct gravity effect of thin film deposition and other crystal growth processes would have far reaching implication both for product improvement and utilization of space environments for processing. Further studies of the gravity effect observed in this study are indicated to elucidate the mechanism and to characterize fully all possible effects.

The present report also reviews some specific candidate ion beam space processing operations.

It might be mentioned that although the present study had as its chief motivation the identification of space processing application for an ion thruster engine, the range of applications of ion beam technology was not limited to the capabilities of any specific apparatus. Thus, applications such as ion implantation are mentioned even though the ion energies required for this task are somewhat higher than produced by a typical TIB generator. It is felt that at this early stage the full range of ion beam technology should be scanned. Possibly a particular application may prove to have enough economic or utilitarian potential to justify a special, high energy TIB design. Also, the state of the art of TIB design will undoubtedly change rapidly as the scientific community becomes more aware of the potential of ion beam processing.

THRUSTER ION BEAM GENERATORS AND GROUND APPLICATIONS

The essentials of a thruster engine are shown in Fig. 1. With a few minor modifications, the engine design is readily adapted for ion beam

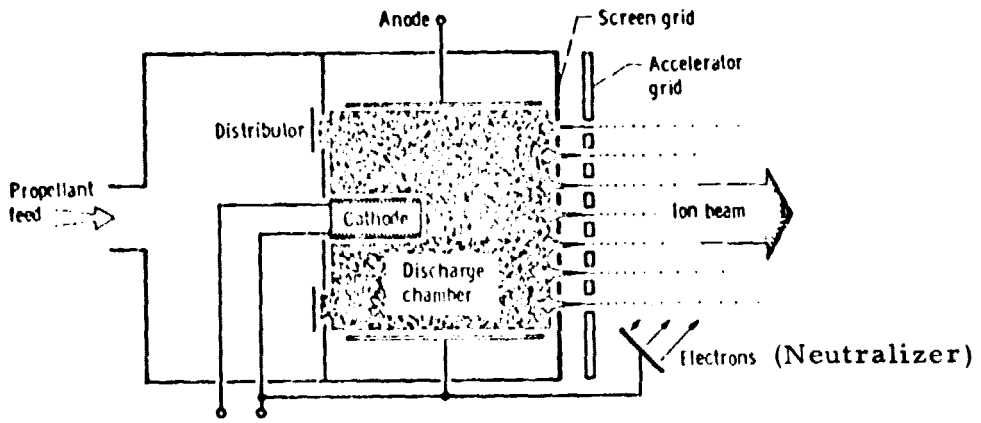
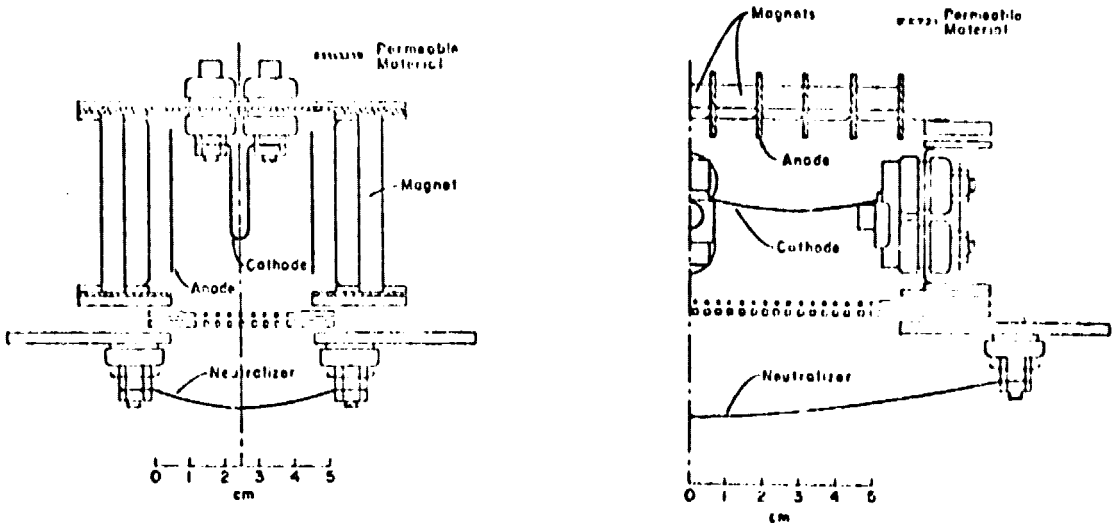


Fig. 1 - Essentials of a Thruster Engine (Ref. 1)

applications. For example, Figs. 2a and 2b show some recent adaptations. Further descriptions of ion beam generators of the thruster type are given in Refs. 2 through 5. Reviews of conventional ion beam generators are given in Refs. 6 and 7. Among the advantages of thruster generators are large beam diameters, precise control of beam size, uniform ion energy, unidirectional ion flow, ease of control, and the contaminant-free, high vacuum



a. Cross Section of 2.5 cm Ion Source b. Cross Section of 10 cm Ion Source

Fig. 2 - Recent Ion Beam Generators Based on the Ion Thruster Principle (Ref. 2)

environment in which the thruster operates (Ref. 3). Some typical characteristics for generators of the thruster type are:

Pressure in Plasma Generator Cavity	$\sim 10^{-3}$ torr
Pressure in Sputter Chamber	$\sim 10^{-4}$ - 10^{-5} torr
Ion Beam Diameters	2.5 cm up to 30 cm
Ion Beam Current Densities	1 - 2 mA/cm^2
Ion Beam Energies	0.5 - 10 keV
Gas Flows (Usually Argon)	~ 1.5 cm^3/min
Acceleration Voltage	0 - 10 kV

It is also possible to design a TIB to eject heavy ions such as mercury (Refs. 1, 8 and 9).

In applications in which materials are processed the ion beam is caused to impinge either on the material itself (cleaning and implantation operations) or on a target material causing a transfer of momentum between the ion beam particles and some of the target particles. The target particles are "blasted"

off the surface, or more commonly sputtered. The sputtered target material is then deposited on a substrate (deposition operation). Figure 3 shows the arrangement for a sputter deposition.

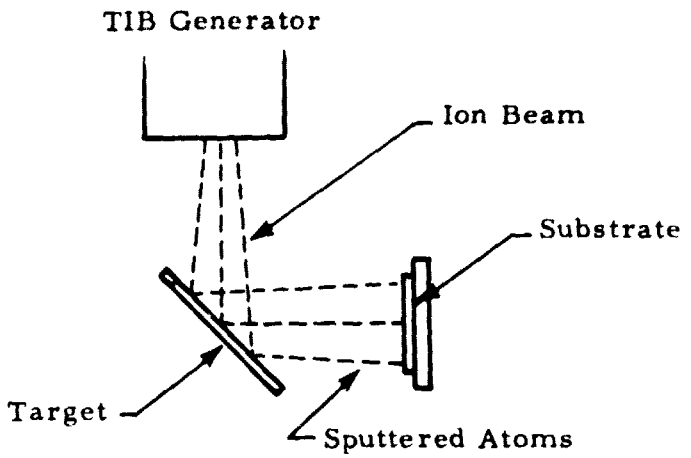


Fig. 3 - Schematic of an Ion Beam Sputter Deposition

The list of actual and potential applications for ion beam technology on the ground is impressive and is expanding rapidly. The following list is indicative.

- Ion Implantation in Semiconductors
- Ion Milling, Etching, and Cleaning
- Deposition of Thin Films of Semiconductors and Metals
- Micro-Circuit Fabrication
- Catalyst Preparations
- Surface and Particle Collision Studies

References 6 and 9 through 19 present good reviews of application in the various areas.

SPACE ENVIRONMENT EFFECTS

A review, with an eye as to possible space advantages of the ion beam applications cited in the preceding section, identified the following common area of benefit: the almost infinite pumping capacity of space environments definitely promises enormous advantages for ion beam technology. Also some exploratory experimental studies of both sputter thin film deposition and vapor deposition indicate that gravity may have a direct role in both processes even at very low pressures. These aspects of the study are presented in the following two subsections.

Advantages of Space Vacuums

In almost all cases of sputter deposition, or vapor deposition for that matter, the quality of thin films obtained depends very sensitively on the degree of gaseous contaminants present during deposition. For example, in the case of germanium thin film deposition, oxygen even at a partial pressure of 5×10^{-9} torr was found to increase epitaxial deposition and amorphous-polycrystalline transition temperatures (Ref. 20). This result was explained on the basis of oxide formation. The great sensitivity of silicon thin film deposition to oxygen and carbon impurities is well known (Refs. 21 and 22). Gaseous contaminants can also influence the quality of metal films (Ref. 23). For example, it has been shown that either β-Ta or bcc Ta may be formed by sputtering at a residual gas pressure of 2×10^{-6} torr and a total argon pressure of 20×10^{-3} torr; but as the residual gas pressure is increased to 5×10^{-5} torr, only bcc Ta is obtained (Ref. 24). Figure 4 shows the results of one study (Ref. 25) on the effects of pressure on tungsten film resistivity. The high resistivity values were attributed to incorporation of residual gases during the growth process.

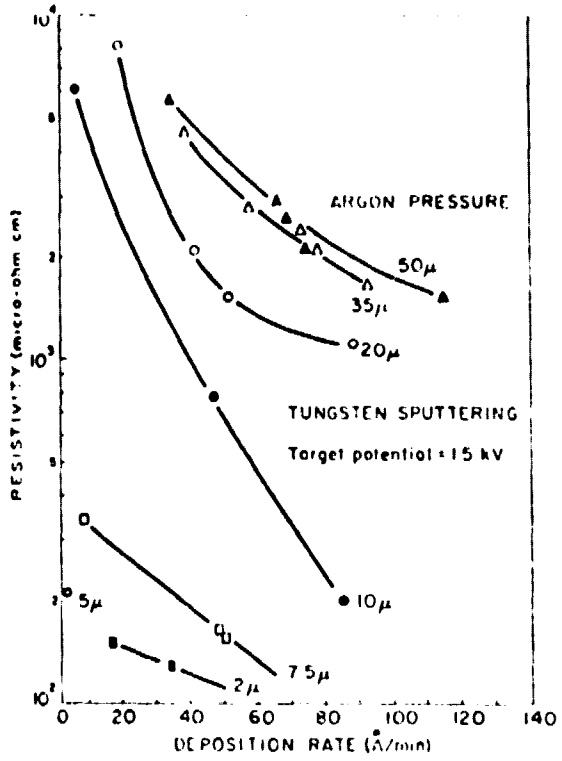


Fig. 4 - Effect of Deposition Rate and Argon Pressure on the Resistivity of Sputtered Tungsten Films (Ref. 25)

The rate of impurity adsorption is also of concern with regard to a sputtering target. Such adsorption can lead to decrease of sputtering, yield and recontamination of surfaces deliberately cleaned. It is stated that the ratio of the ion current density ($\mu A/cm^2$) to the background pressure (torr) should be greater than 10^8 to avoid formation of surface layers (Ref. 26). Obviously, the lower the background pressure the better.

The impingement rates (n, number per cm^2 per sec) of atoms at various pressure levels can readily be calculated from the following formula (Ref. 27)

$$n = 3.51 \times 10^{22} P/\sqrt{MT}$$

where P is the residual gas pressure in torr, M is the molecular weight, and T is the absolute temperature. The maximum impurity content, K, due to

residual gases in a sputter deposited film is given by the following formula (Refs. 27 and 28):

$$K = \frac{5.82 \times 10^{-2}\, \alpha\, MP}{\rho\, dD/dt \sqrt{M'T}}$$

where

α = sticking probability of the residual gas on the fresh film surface

M = molecular weight of thin film material

M' = molecular weight of gas

T = absolute temperature of gas

ρ = film density

dD/dt = deposition rate

P = gas pressure, torr

Table 1 gives K values for various levels of oxygen pressure and various rates of Mo deposition ($\alpha = 1$).

Table 1

K VALUES FOR VARIOUS MOLYBDENUM DEPOSITION RATES AND OXYGEN PRESSURES (REF. 28).

p of Oxygen (torr)	Deposition Rate (Å/s)		
	1	10	100
$1.6.10^{-9}$	10^{-3}	10^{-4}	10^{-5}
$1.6.10^{-7}$	10^{-1}	10^{-2}	10^{-3}
$1.6.10^{-5}$	10	1	10^{-1}

Thus, even at an oxygen pressure of about 10^{-7} torr a molybdenum layer deposited at a rate of 10 Å/s can contain 1% oxygen. For many films this level of impurity is much too high.

Two factors are common to any type of sputtering system: both the background pressure and the throughput of sputtering gas must be maintained at some predetermined levels. The preceding formula for K indicates that

control of impurity level can thus be controlled in a film deposition by one of three means: reducing α (bias sputtering), increasing the deposition rate, or reducing the partial pressure, P_i, of the impurity. The partial pressure of an impurity is related, of course, to the purity of the sputtering gas. Further, the partial pressure of impurity is related not only to the total pressure but also to the rate of system leakage. The partial pressure of a contaminant is therefore related to the throughput of sputtering gas. The formula relating these quantities is given by the following (Refs. 29 and 30):

$$P_i = LP/(Q + L)$$

where L is the related leak rate and Q the throughput of sputtering gas in torr liters/sec. Leaks may arise from a number of different sources — actual leaks in the apparatus, desorption from apparatus walls, "backstreaming," and diffusion of gas through the walls of the apparatus (Ref. 31). Of these different sources of leaks, desorption and "backstreaming" appear to be the most serious (Refs. 31, 32 and 33). The preceding formula relating impurity partial pressure to leak rate and throughput rate indicates that it is advantageous to make the throughput as large as possible and the leak rate as small as possible. The throughput rate is related, of course, to the pumping speed.

The optimum pumping speed required to maintain a total pressure of argon of 10^{-2} torr and a throughput of 1 torr liter/sec is a few hundred liters per second (Ref. 28). Based on this criterion sputter-ion pumping in earth atmosphere is judged as too cumbersome and expensive; standard sorption pumps are inadequate because of small sorptive capacity; reproducible vacuum conditions are hard to achieve with cryopumping; diffusion pumps involve complications due to oil vapor backstreaming; and turbomolecular pumping, the recommended pumping method, is somewhat expensive and also can involve complications due to water, hydrogen, and helium backstreaming (Ref. 28). Backstreaming problems with turbomolecular pumps was the topic of lively discussions in the recent literature (Ref. 34).

The preceding discussion of pumping speeds relates to conventional type sputtering systems. In the ion beam method, the ion beam is generated at about 10^{-3} torr and the beam is then directed into the work chamber which is at a pressure of about 10^{-5} torr. Under such conditions, it is expected that the requirements on pumping speed will be even more severe, especially with ion beams of wide area and high current density. For example, it is stated that present commercial ion beams with current densities of about 1 mA/cm^2 (total currents about 15 to 50 mA) will typically have gas flows of 1.5 cm^3/min (Ref. 35). This gas flow corresponds to a throughput of 1.9×10^{-2} torr ℓ/sec and would require a pump with pumping speed of 1900 ℓ/sec to maintain a pressure of 10^{-5} torr. If leak rates were taken into account the pumping rate would have to be higher still. In one instance in which an apparatus was so designed that an ion current density of 50 mA/cm^2 (total current 350 mA per about 7 cm^2 outlet area) was obtained, a pumping speed of 2000 ℓ/sec was insufficient to maintain an order of magnitude pressure difference between the source and the background. The pressure in the source had to be dropped to 5×10^{-4} torr (Ref. 36). Because of lack of practical, inexpensive systems that can pump at more than about 2000 ℓ/sec, ion beam is generally limited at present to depositions in the range of about 10^{-5} torr or higher (Ref. 37). The advantage of higher pumping speeds, i.e., higher throughputs, would be that greater beam areas and higher beam current densities could be obtained (because of reduced charge-exchange and background scattering effects (Ref. 2)) and film deposition or sputtering could occur at even lower pressures (less contamination).

In view of the problems of obtaining adequate pumping speeds with various pumps, the advantages of utilizing the natural vacuum of space becomes intriguing. The pressures at various altitudes are generally as follows: 60 km – less than 1 torr, above 600 km – less than 10^{-8} torr, and above 1200 km – less than 10^{-10} torr (Ref. 38). In outer space pressures of 10^{-13} torr and less prevail. Although the statement is often made that the pumping speed of the space vacuum is essentially infinite (Ref. 39), a more accurate statement is that the near-vacuum environment of space affords a readily usable pumping capability of, for all practical purposes, ultimate efficiency. Only the "induced" atmosphere of the spacecraft and its associated hardware and instrumentation would detract

from the usability of the space environment as a vacuum source. This induced atmosphere may originate from a number of sources, such as drifting gas, outgassing of spacecraft materials (paints, sealants, adhesives, etc.), dumping of waste products and firing of attitude control motors. These various considerations have recently been analyzed for the purpose of assessing the feasibility of a molecular shield for the Space Shuttle Orbiter (Refs. 40 through 43). The concept of the molecular shield is shown in Figs. 5 and 6.

The analyses indicate that pressures of about 10^{-14} torr can be easily achieved behind the molecular shield. Given a proper orientation of an ion beam apparatus behind the molecular shield, it should be possible to attain an almost infinite pumping speed. Further advantages of the high vacuum behind a molecular shield are that larger systems could be handled than on earth. Costly hold-up times for degassing and baking operations could be avoided. Also not to be ignored are the initial vacuum system cost, cost of coolants, and cost of required power input, which apparently can run from about 0.2 to 22 kW depending on the particular pumps (Ref. 44). The latter factors of cost and convenience will probably become of more and more importance as the trend toward sputter utilization and automated systems continues to grow (Ref. 45).

Gravity Effects

A number of experiments on thin film deposition were conducted in the course of the present study. The results of these experiments indicate that gravity may be a significant variable that has been hitherto overlooked. The evidence indicating possible gravity effects in high vacuum thin film deposition are reviewed in the following paragraphs.

The ion beam facility at NASA-Lewis was utilized to deposit simultaneously a number of thin films of copper and sapphire at a background pressure of 10^{-6} torr. The physical arrangement employed is shown in the sketches of Fig. 7.

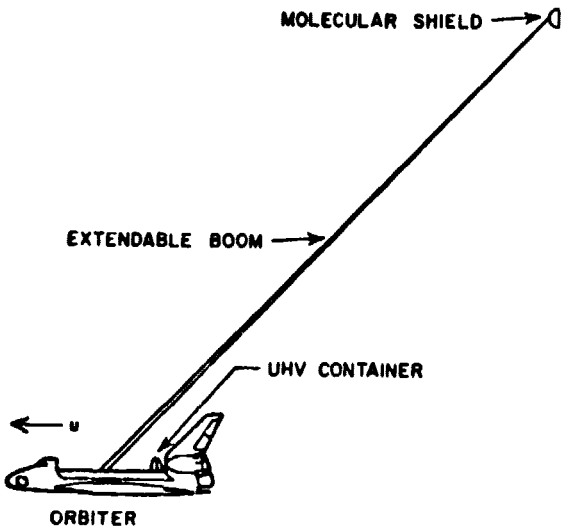


Fig. 5 - The Molecular Shield Shown Deployed from the Space Shuttle by an Extendable Boom. The length of the boom will be determined by the magnitude of the density around the Shuttle (Ref. 42)

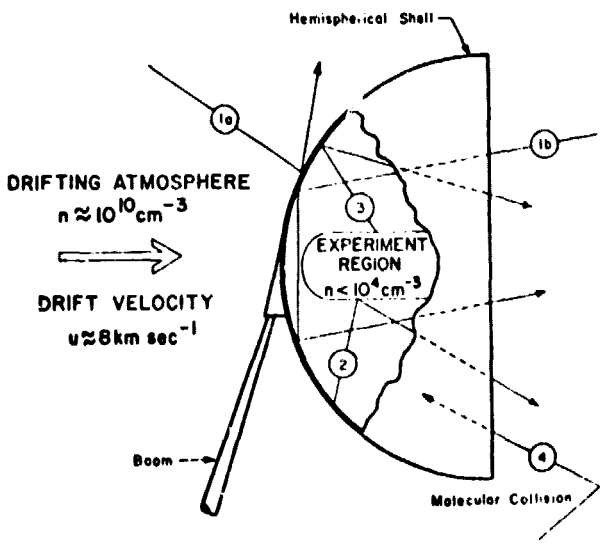
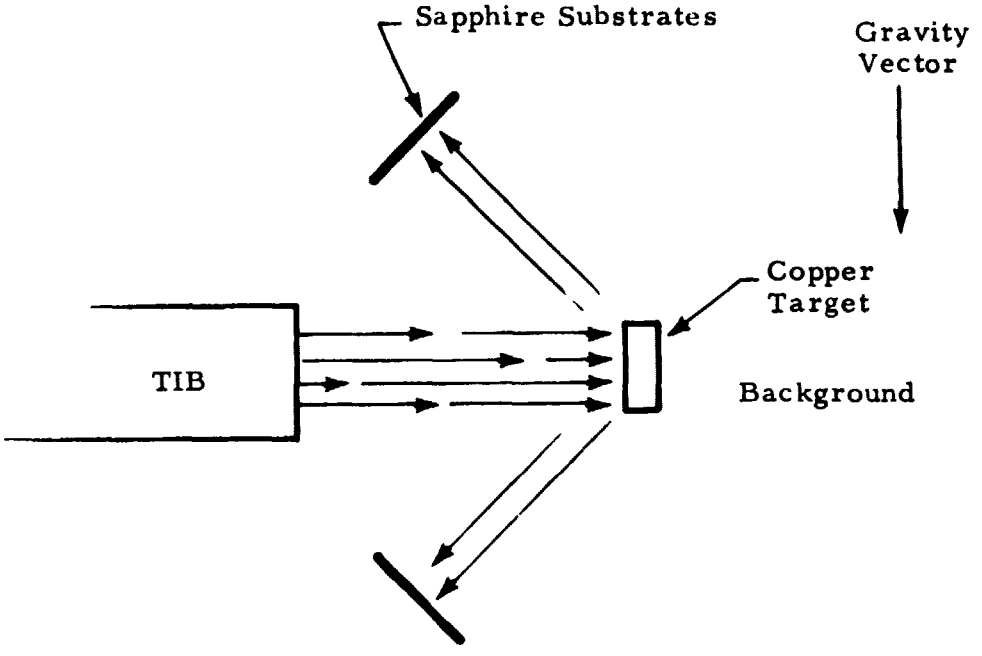
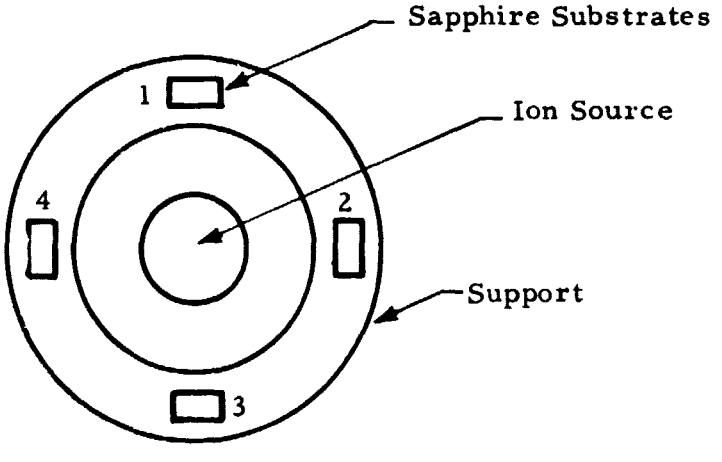


Fig. 6 - Schematic Representation of the Molecular Shield Geometry in the Drifting Gas, Illustrating Typical Molecular Trajectories; (1a) and (1b) free stream molecules, flux of (1a)-type molecules is much greater than flux of (1b)-type molecules; (2) desorbed molecules from the shield; (3) desorbed molecules from the experiment; and (4) molecules scattered from the orbiter (Ref. 40)



a. Side View



b. Head-On View of Substrates

Fig. 7 - Experimental Arrangement for Sputter Deposition of Copper or Sapphire

The ion beam consisted of argon ions and atoms. Figure 8 shows the differences in the structures of the thin films deposited in the noted positions. It is difficult at present to make much of the noted differences. A number of further experiments are needed in which variables such as ion beam engine stability, exact sample location, and impurity levels are rigoriously controlled. Evaporative depositions which are more easily controlled, however, do indicate a gravity effect, as described in the following discussion.

The works of Sherman, Mao, and Hutchinson (Ref. 46), Chopra (Ref. 47), Lane and Anderson (Ref. 48) showed relatively slight differences in thin film structures as the result of method of deposition (sputter or evaporation). These differences were attributed to different surface mobilities because of the different initial kinetic energies and different electrostatic charge environments. Thus, it is probably safe to assume that a gravity effect observed in a vacuum evaporation deposition would also apply to a similar sputter deposition. In a series of evaporative-deposition laboratory tests numbering about fifty and utilizing model organic materials, differences as the result of the position of the substrate with respect to gravity were also noted. The two experimental arrangements employed are shown in Figs. 9 and 10. The organic materials utilized in the evaporative tests included camphor, biphenyl, and acetamide. Glass, sapphire substrates, and plastic substrates were also utilized and were cleaned by a variety of techniques prior to crystal depositions. The temperatures were moderate (~ -10 C to 25 C for substrates, and ~47 C to 90 C for sources) and were not monitored at the substrate surfaces. The temperatures, however, were at the same values for any given top/bottom run couple. Differences in crystalline deposits depending on whether the substrate was on the top or the bottom relative to the source were noted in all but two or three cases. In general more numerous, smaller crystals were observed on bottom substrates than on top substrates. The crystalline forms observed on top substrates were larger and tended to be more dendritic than those on bottom substrates. Dendritic type crystals of biphenyl and camphor would at times be quite curled. Curled dendrites when they occurred were more prevalent on top substrates than on bottom substrates. In the case of acetamide at about 1 torr the differences are quite dramatic: the top substrate shows a few large needles extending out from the substrate surface, while the

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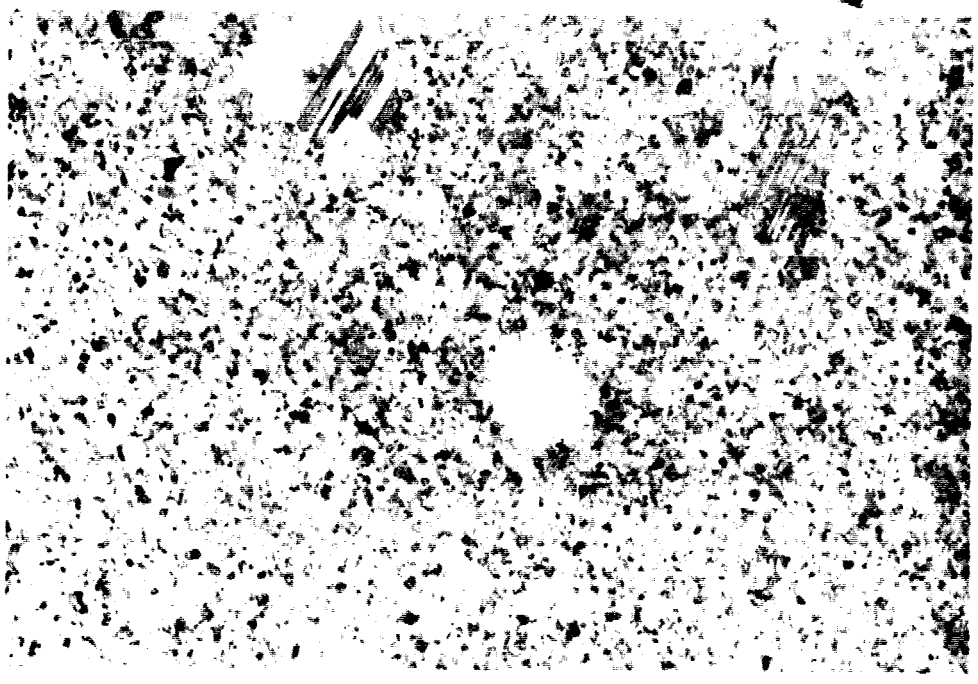
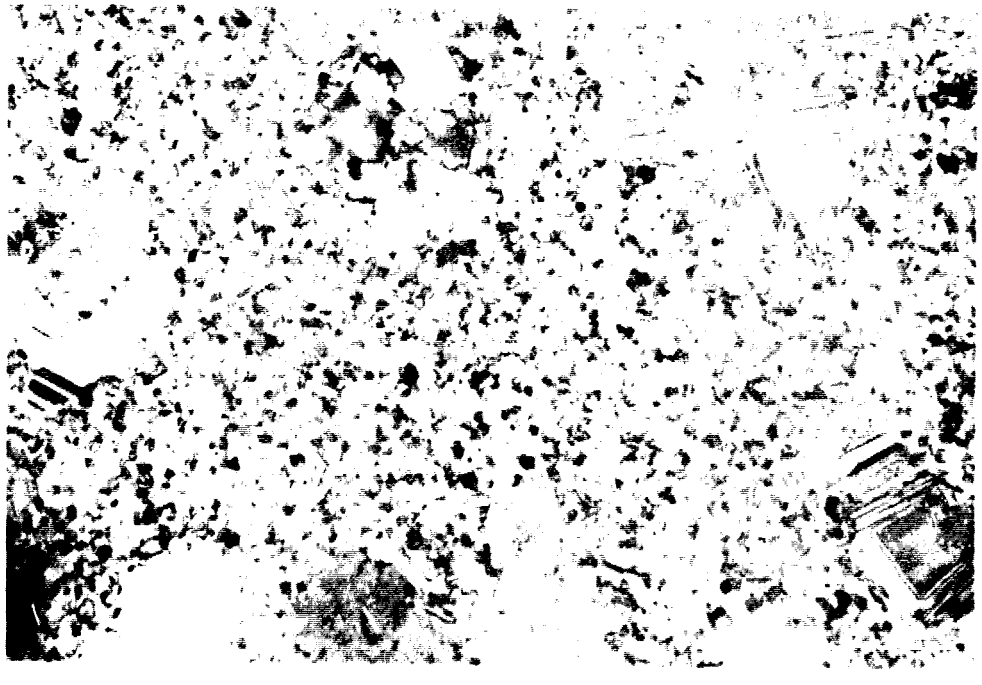


a. Deposit on Top Substrate



b. Deposit on Bottom Substrate

Fig. 8 - Electron Transmission Micrographs of Copper on Sapphire Deposits

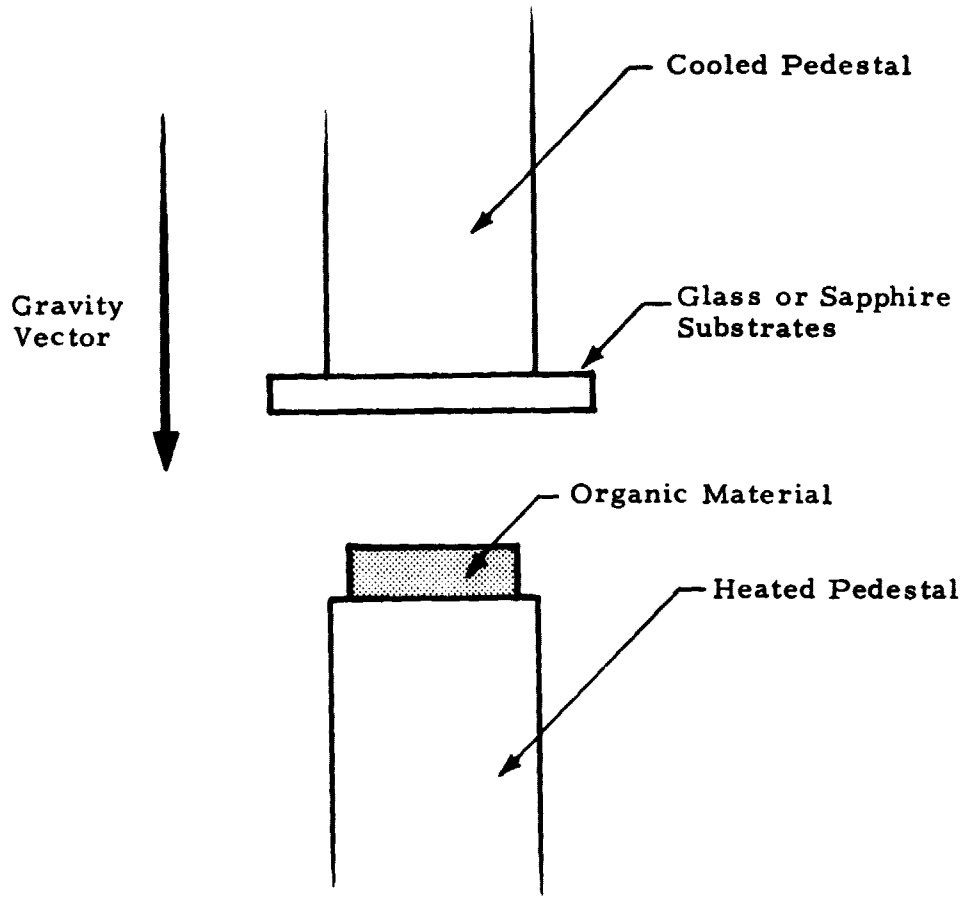
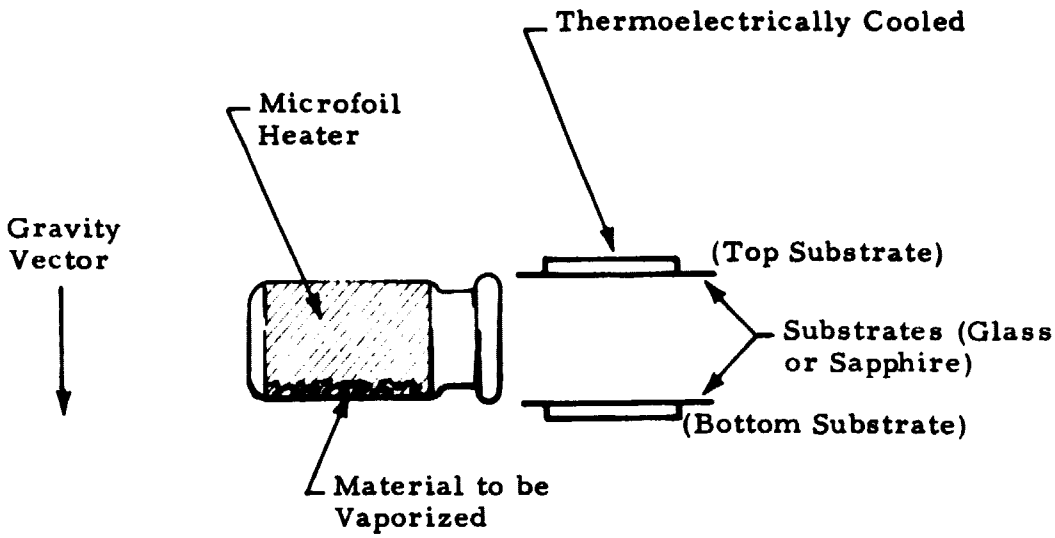
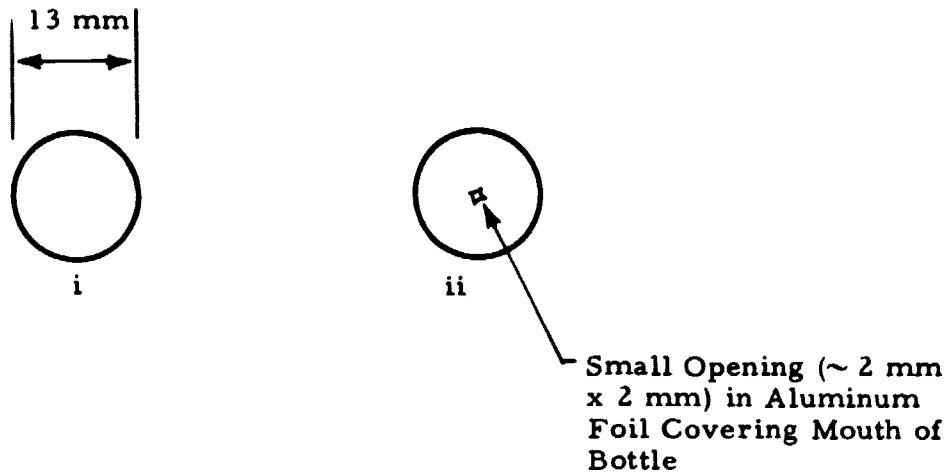


Fig. 9 - Apparatus Used at 1 atm Pressure in Upright Position (Further tests were conducted with the apparatus inverted. A nylon net or fiberglass web over the organic source material prevented the material from falling out.)



(Widest Diameter of Bottle ~ 19 mm, Height ~ 34 mm)

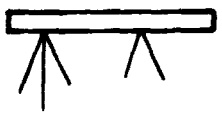
a. Side View



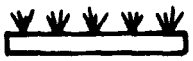
b. Head-On Views of Heated Container Openings

Fig. 10 - Apparatuses Used at Vacuum Levels of About 1 torr

bottom substrate shows many small needles, also extending out from the surface, as shown in Fig. 11.



Acetamide Deposit
on Top Substrate



Acetamide Deposit
on Bottom Substrate

Fig. 11 - Crystalline Deposits Obtained in Low Pressure Evaporative-Deposition Tests

In the evaporative-deposition tests variables such as substrate cleaning procedures, size of source opening in the low pressure tests, and possible differences between substrate coolers were checked and found not to be the cause of the observed effects. Also in the case of the low pressure apparatus a series of tests were performed in which the heater power was reduced in order to reduce the evaporation temperature. Longer deposition times were utilized in these reduced temperature runs. Definite differences between top and bottom deposition were still noted, although the differences were less marked than in the higher heating rate cases. It might be added that two different experimenters conducted the various tests and did not compare notes on the structures observed until most of the work was completed.

The evidence indicates that gravity can affect crystal growth even at low pressures. More extensive tests on both ion-beam sputter deposition

and high-vacuum evaporative deposition of metal and semiconductor materials are required to absolutely substantiate, however, this startling conclusion.

Discussion: A consideration of possible causes of the observed behavior other than direct gravity effects elicited two — gravity-driven convection or directional effects related to mean free paths. Neither of these possible causes, however, can be supported for the following reasons. In order for convection to have been the cause of the observed results it would have been necessary that vigorous convection was present at 10^{-6} and 1 torr as well as at 760 torr (1 atm). The vigor of gravity-driven convection, however, decreases rapidly with decreasing pressure. The thermal and solutal Grashof numbers, which are the ratios of the buoyancy forces generated by thermally or solutally induced density differences to the viscous forces and hence may be viewed as indicative of convective vigor, are related to pressure by the following relationships:

$$Gr_T = \frac{g d^3 \Delta T}{T \nu_o^2} (P/P_o)^2 = Gr_T^o (P/P_o)^2 \qquad \text{(Ref. 49)}$$

$$Gr_s = \frac{[M_B - M_A] g d^3 \Delta S_A}{\rho_o \nu_o^2} P/P_o \qquad \text{(Derivation given in Appendix A)}$$

where

- g = gravity acceleration
- d = a characteristic distance across which the temperature or concentration difference is composed
- ν_o = kinematic viscosity at a reference pressure
- P_o = reference pressure (1 atm usually)
- ΔT = temperature difference across characteristic distance
- ΔS_A = concentration difference of component A across characteristic difference, concentration here given is in weight fraction units
- ρ_o = density at a reference pressure
- M_A, M_B = molecular weights of solution components A and B

The formula relating the thermal Grashof numbers to pressure shows that the Grashof numbers, and hence the vigor of natural convective flows, decrease rapidly with decreasing pressure and becomes quite small at pressures of 1 torr (133 Pascals) and very small at 10^{-6} or 10^{-7} torr (10^{-4} or 10^{-3} Pascals). At atmospheric pressures the Grashof numbers will typically be on the order of 10^{2} to 10^{5} (weak to vigorous convection). At one torr these numbers would be reduced to 10^{-4} to 10^{-1} and to 10^{-16} and 10^{-13} at 10^{-6} torr. The huge differences in Grashof numbers at the three different pressures used in the present study makes it very unlikely that gravity-driven thermal convection could have been the cause of the observed results. This conclusion finds support in an experimental study of convection in tall, thin layers of air uniformly heated at one wall and cooled at another (Ref. 50). The thickness of the interlayer was varied 5, 10, 15, 20 and 25 mm. The temperature drops between the heated and cooled walls was also varied, i.e., ΔT = 20, 40, and 60 K. It was found that convection becomes negligible below 10 torr.

From the formula for the solutal Grashof number, it can be surmised that pressure will not reduce convection vigor nearly as much in the case of solutal convection for a given concentration difference as it does in the case of thermal convection for a given temperature difference. At 10^{-6} torr, however, any concentration difference is bound to be very small. There are about 3.5×10^{10} particles per cubic centimeter corresponding to a concentration of about 5.9×10^{-11} moles per cubic centimenter or 5.9×10^{-8} molar at a pressure of 10^{-6} torr. A concentration difference of 50% would still be only 3.0×10^{-8} molar. It appears, therefore, that neither solutal convection nor thermal convection could be a cause of the observed effect.

The possibility that directional effects related to mean free paths could have been a factor also does not seem likely. The mean free paths at 1 atm and at 1 torr are 2×10^{-5} cm and 5×10^{-3} cm. The distances between the evaporation source and the substrates in the 1 atm and the 1 torr cases were about 1 and 2 cm. Also, in the sputtering experiment directional effects could not have been a factor because both the top and bottom substrates were equidistant from the target.

One possible explanation of the observed results might be that small crystals are nucleated in the vapor phase just above the substrate. Depending on the direction of gravity then, they may either fall onto or away from the substrate. This view finds some support in a couple of literature reports. A nucleation mechanism in the gaseous phase was postulated as the cause of rough surfaces obtained in the growth of silicon epitaxial layers (Ref. 51). In a recent study of frost deposition some secondary growth of small particles between the primary growing crystal stalks is shown (Ref. 52). The strongest bit of support for this explanation is the paper by Horodecki et al. (Ref. 53) in which a gravitational difference was found in the chemical vapor transport growth of ZnS crystals. The difference was attributed to the settling of heavier crystallites in the gravitational field. The formation of secondary crystallites in high vacuum depositions would necessitate some sort of a loose adsorption layer of adatoms. The prospect is intriguing but further speculation is unwarranted. Further investigations of both high vacuum sputtering and vapor deposition are needed to more fully establish and characterize the gravity effect.

SPACE APPLICATIONS OF ION BEAM TECHNOLOGY

High Quality Thin Films and Unique Catalysts

The previously mentioned fact that many semiconductor and metal thin film processes are extremely sensitive to small amounts of impurity atoms is a major consideration in contemplating potential space processes. In particular, the possibility that large scale processing of silicon thin films by a TIB process in vacuums of 10^{-6} torr or better offers the promise of a means by which cheaper solar cells can be produced. It would also not be surprising that oxygen or other impurities play a major role in the ion beam sputter deposition of unique films such as diamond (Ref. 54), superconductors (Refs. 55, 56, and 57) and amorphous bubble domain films (Ref. 58). The more precise control of impurity level afforded by the larger pumping capacities available in space environments would facilitate better quality films at lower cost.

Two areas of catalyst production appear to have some promise: Formation of very small particles and subsequent deposition on an inert substrate either by a means such as ionized particle beam method (Ref. 59) or the more conventional sputter deposition. The second area that appears promising is that of co-deposition of metal atoms with vapors of organic or inorganic compounds at low temperatures (Ref. 60). The present method used to produce such compounds is a vapor deposition process. It appears that an ion beam deposition would offer a more economical procedure. The advantage of space for such processes would be the unlimited vacuum and possibly lack of a direct gravity effect on particle size and morphology would result in unique structures.

Complex Fabrication Operations

The most compelling promise of ion and electron beam processing in space is that of economics and product improvement realized from being able

to completely automate large-scale production operations because of the large pumping capacity available in space. For example, a future space fabrication utilizing TIB might consist of the following:

a. Sputter deposition of semiconductor thin film
b. "Writing" a circuit on the thin film by means of a focused ion beam (Refs. 6 and 13)
c. Sputter depositing passivating layers of SiO_2 (Ref. 61).

The following quote, although written as a prediction of the probable direction of microelectronic fabrication processes on earth, is probably just as accurate as a prediction of the direction microelectronic fabrication in space will take.

> "Electron beam pattern fabrication combined with the complementary ion beam processes offers some unique features; some very useful devices just cannot be made in any other way! The potential yield improvement by all-vacuum processing, using electron and ion beams, together with automated electron beam testing, presents goals worth working toward. Beams are presently not widely used or fully developed, and involve relatively sophisticated processes using generally unfamiliar and expensive equipment. Nevertheless, in the writer's opinion, they form the next major process technology for microelectronics. In some cases beam processes may replace contemporary fabrication processes; more often they will be applied in ways that take best advantage of their unique features."
>
> — G. R. Brewer (Ref. 11)

Construction of Large Structures in Space

A number of concepts are currently under study for construction and utilization of large space structures. Among such potential large space structures are large arrays for converting solar energy into microwave energy which would then be beamed to earth; platforms for large space telescopes; platforms for earth observations; experimental stations, and space colonies. It is already generally predicted that it will be more economical to perform as much as possible of the construction in space rather than on earth. Also, it will probably be more economical eventually to obtain the required materials from the moon rather than from the earth. These eventualities

will require processing techniques uniquely suited to the space environment. TIB appears ideally suited to perform a multitude of tasks associated with construction of large space structures. Some of these predicted uses are presented in the following subsections.

Cold or Warm Welding: The suggestion was made by Bernard Sater of NASA-Lewis of utilizing the ion beam in space for the purpose of cleaning the surfaces of pieces preparatory to cold welding. The possibility of ion beam cold welding is intriguing because it appears to hold many advantages for construction of large space structures in the vacuum, and weightlessness environment of space.

On earth processes such as diffusion, cold, and explosive welding are fairly sophisticated techniques utilized to limited extents. These methods require careful surface and edge preparation and fit-up and large specific pressures (Ref. 62). Electron beam welding which is also a relatively sophisticated technique, on the other hand, is winning widespread acceptance for earth applications. Electron beam welding in space, however, appears to have several disadvantages. For example in a space test of electron beam welding of aluminum alloy AM-6, the space welded samples were more porous than the earth welded samples (Ref. 62). Porosity problems which are frequently encountered on earth (Refs. 64 through 66) therefore, will probably be intensified in space. Electron beam welding, in common with other conventional methods of welding, necessitates severe heating and melting in the areas of the welds. Severe stresses, therefore, may be generated, resulting in weakened structural capabilities. Still another factor to be considered are the power requirements for electron beam welding, especially if wide welds are required. A weld width of 0.127 cm might require a total power of 25 kW (Ref. 64). Also not to be ignored is the fact that high energy electron beams can generate appreciable x-rays (Ref. 67). Astronaut exposure would therefore present shielding problems.

Relatively little research has been conducted on cold welding methods. References 68 through 77 present some reviews of the recent work and

experience with cold welding. Very little research also has been thus far conducted on ion beam cold or warm welding. (The term warm welding was coined for contact welds made at temperatures of about 0.1 to 0.51 T_m where T_m is the melting temperature, degrees K (Ref. 78). A number of papers, however, have considered the mechanisms of cohesion and adhesion (cohesion – a joining of pieces of the same material; adhesion – a joining of pieces of dissimilar materials) and the effects of surface conditions (Ref. 79 through 82). The two main ideas incorporated in various theories of cohesion/adhesion bonding mechanisms are those of contaminating film presence and energy barriers due to causes such as misorientation of crystals at the surface or recrystallization processes (Ref. 68). It is doubtful that the effects of film presence on dislocation movement, and hence on subsequent plastic flow and bonding, are completely understood. It does appear, however, that the following can be cited as prerequisites for a good cold or warm weld; clean surfaces (free of oxides or other contaminants), a certain degree of surface roughness, sufficiently ductile materials (high stacking fault energy), and a certain degree of thermal activation with some materials. In the case of very hard solids, however, it is claimed that surface roughness can greatly reduce the adhesion between solids (Ref. 80). It might be mentioned that in the two studies that dealt specifically with ion plasma cleaning of the surfaces prior to surface contact (Refs. 78 and 83) successful welds were produced with a number of materials.

From the viewpoint of ion beam cleaning of surfaces and subsequent welding in space, the following advantages are postulated.

- Easy, fast cleaning of surfaces of large extent
- The ultra high vacuum of space, especially if molecular shields are provided, would ensure very clean surfaces for relatively long periods of time
- Relatively low energy requirements
- Control over the surface roughness
- Control over degree of thermal activation.

Because of the cited advantages it is foreseen that joining large structures in space would be facilitated by the technique. Joining materials such as composites, metal-ceramic or metal-semiconductor materials should also be better accomplished by ion beam than with other existing techniques.

Another aspect of ion beam technology with regard to cold welding is that of sputter deposition of an intermediate film that would make possible the joining of two very dissimilar materials. References 70 and 84 list some composites that have been successfully joined by cold or diffusion welding.

Strenghtening Construction Glass: It is predicted that because glass will be one of the materials most readily obtained from the moon, glass will be utilized to a great extent in space as a construction material. One of the problems with glass as a construction material to date has been its low tolerance for tensile stress. Recent work, however, indicates that this tolerance can be increased by putting the surface into compression because glass can withstand almost infinite compressive stress. One means of doing this is by the replacement of glass alkali ions by larger alkali ions present in a chemical bath (Refs. 85 and 86). It would seem that ion beam sputtered films or implantation could be used to accomplish the same task more rapidly and economically. Apparently the National Bureau of Standards has accomplished some work along these lines (Ref. 87).

Coating, Texturing, and Milling Operations: A number of coating operations will of necessity be conducted in various constructions, i.e., reflective, protective, and decorative coatings. Wide area ion beam sputtering should be well adapted to such tasks. The formation of unique surface textures by means of sputter etching, i.e., "cone" formation (Ref. 88) could find application in space for absorptive solar collector panels. The only question in this regard is the advantages ion beam might offer over other techniques. It would seem that lesser energy expenditures and greater control over coating thicknesses would be the advantages of ion beam depositions. Also, the possibility of combining evaporation and ion beam sputtering could lead to further advantages, i.e., ion plating as shown in Fig. 12. In the area of milling operations in space, of course, ion beam appears to have no competitors.

Very High Energy Heavy Ion Beam Applications

In this subsection some applications of very high energy ion beams are reviewed. Although TIB technology has not been involved in generating very

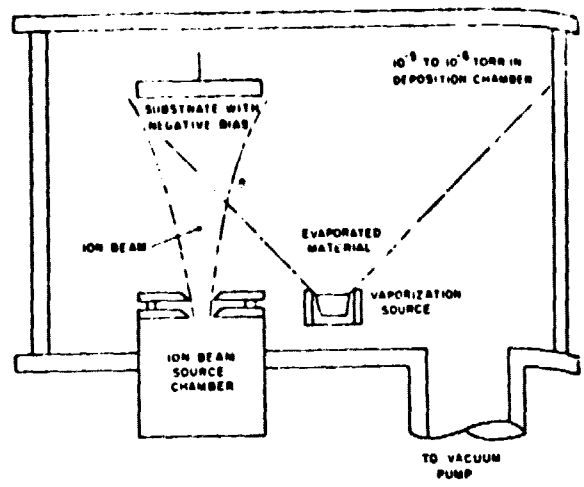


Fig. 12 - Illustration of Vacuum "Ion Plating" Technique Using a Vaporization Source for Fast Deposition and an Ion Beam Source to Supply Energy and Momentum to the Film Atoms for Improved Film Properties (Note that the use of high vacuum conditions improves the film properties (Ref. 89).

high energy ions beams (~160 MeV - 10 GeV), the topic is brought up here because it is related to TIB. It would seem that the generation of very high energy ion beams would also benefit from space environments because of the "free" vacuum and high pumping capacity conditions. Producing very high energy, heavy ion beams on earth is a very costly business (Ref. 90). Such beams are primarily of interest for fundamental studies and possibly for fusion power (Ref. 91). Some further novel applications, however, have been suggested (Ref. 9): modeling of radiation damage in reactor materials, production of filters of ultrasmall size and quality (such filters would have many unique uses, i.e., drinking water free of bacteria under field conditions, filtration of aerosols, etc.), material surface studies, radiotherapy to very limited portions of the human body with minimal damage to surrounding tissue, "ionic" surgery, and production of artificial isotopes.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

Space environments afford almost limitless pumping capacities. A limitless pumping capacity will allow easy maintenance of high pressure differentials and thus will allow optimal conditions for operation of numerous ion beams simultaneously or for ion beams of large area. The easy maintenance of a large pressure differential will further allow thin film deposition under conditions of ultra-high vacuum. Such advantages recommend large-scale utilization of ion beams for operations such as microelectronic component fabrication, which would result in product quality improvement at lower cost.

Experimental studies of both ion beam sputter and vapor deposition of thin films indicate a direct gravity effect on the crystallization processes even at high vacuum. This direct gravity effect could have enormous, far reaching implications both for product improvement and utilization of space enviroments for processing.

Further space processing applications of ion beams include: welding; strengthening of construction glass; and coating, texturing, and milling operations. Possibly higher energy TIB will eventually be designed. Space processing applications of higher energy beams might include production of filters of ultrasmall size and quality and production of artificial isotopes.

Recommendations

An analysis of the economics of producing a particular thin film (silicon for example) by means of ion-beam-sputter and by vapor deposition under ultrahigh vacuum conditions both on earth and in space is recommended.

Also recommended, to conclusively establish the direct gravity effect and its mechanism, are further experimental studies. Particularly needed are further ultrahigh vacuum depositions employing both ion beam sputter and vapor techniques. A variety of metal and semiconductor materials should be investigated.

ADDENDUM

After the present report was completed, some further electron microscope transmission micrographs pictures of copper films deposited by ion beam sputter deposition were received. These pictures were of thin films deposited in the 2 and 4 positions as shown in Fig. 7b. The pictures received were very similar to those shown in Fig. 8 indicating that the experiment needs to be repeated with more rigorous controls on ion beam stability, impurity levels, and sample placement.

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Appendix
DERIVATION OF A SOLUTAL GRASHOF NUMBER

Appendix

The derivation of the pressure dependency of the solutal volumetric expansion coefficient is unclear in the literature. The following derivation was obtained in the present study.

$$PV = (n_A + n_B)\, RT$$

where n_A and n_B are moles of A and B, and

$$P = (S_A + S_B)\, RT$$

where S_A and S_B are concentrations (moles/unit volume). Furthermore,

$$\rho = MA\, S_B + MB\, S_B$$

$$P = \left(S_A + \frac{\rho}{MB} - \frac{M_A}{MB} S_A\right) RT$$

$$0 = \alpha S_A + \frac{\alpha\rho}{M_B} - \frac{M_A}{M_B}\alpha S_A$$

$$-\alpha\rho = M_B\, \alpha S_A - M_A\, \alpha S_A = (M_B - M_A)\, \alpha S_A$$

$$-\frac{1}{\rho}\left(\frac{\alpha\rho}{\alpha S_A}\right)_{P,T} = \frac{(M_B - M_A)}{\rho}$$

This result was first given by Gebhart and Pera (Ref. 92).

Following Ref. 49 it is easy to show that

$$Gr_s = \frac{[M_B - M_A]\, g\, d^3\, \Delta S_A}{\rho_o\, \nu_o^2} \cdot P/P_o = Gr_s^o\, P/P_o$$